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14. ABSTRACT

This document summarizes the results of 3 and 1/2 years of work by our team on the basic physics and technology required for the development of Colloid Thrusters (also called more recently Electrospray Thrusters). The research covered the basic physics of operation in the mixed ion-droplet mode, later extended to the newly discovered pure ionic mode, the microfabrication in Silicon of two types of arrays of colloid or electrospray emitters, and the development of a quantitative theory for the colloidal regime (no ions). This Executive Summary describes briefly the various achievements, with reference to our interim Reports and to our publications. Special emphasis is placed on the results that were obtained during the one-year extension (2004), which were not covered in our interim reports. The Theses, published papers and Conference papers for 2004 are enclosed in a companion CD disk; earlier Theses and publications were similarly attached to previous Reports.

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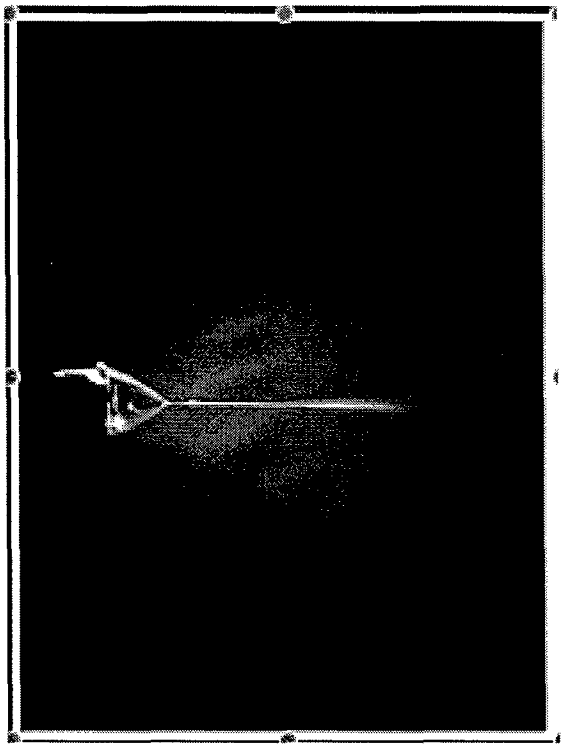

Final Report

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Colloid Thrusters & Physics, Fabrication and Performance

Abstract

This document summarizes the results of 3 and 1/2 years of work by our team on the basic physics and technology required for the development of Colloid Thrusters (also called more recently Electrospray Thrusters). The research covered the basic physics of operation in the mixed ion-droplet mode, later extended to the newly discovered pure ionic mode, the microfabrication in Silicon of two types of arrays of colloid or electrospray emitters, and the development of a quantitative theory for the colloidal regime (no ions). This Executive Summary describes briefly the various achievements, with reference to our interim Reports and to our publications. Special emphasis is placed on the results that were obtained during the one-year extension (2004), which were not covered in our interim reports. The Theses, published papers and Conference papers for 2004 are enclosed in a companion CD disk; earlier Theses and publications were similarly attached to previous Reports.

1. Basic Physics (6/01-11/03)

Our work in this area began almost simultaneously with the commissioning of our DURIP-funded experimental facilities, in particular our small (18''x36") vacuum tank and ancillary instrumentation, that was almost entirely dedicated to this program. Our initial efforts[1] were related to the development of a reliable Time-of-Flight (TOF) and energy analysis setup. The following two years[2] saw the culmination of our experimental work on highly conducting solutions operating at very low flow rates so as to generate an abundance of ions as well as charged droplets. An attempt was made to achieve conditions where the droplet component would disappear altogether (a regime that appeared possible in principle). However, this purely ionic regime has been so far found to occur only when using a different kind of liquid (Ionic Liquids, or Organic Molten Salts), as was documented first at Yale U. in a companion program [3], and quickly verified in our Laboratory. The mixed regime holds some interest by itself, since the mean mass velocity of the spray (ions + drops) can be tailored to yield a wide range of specific impulses (from a few hundred seconds to about 2,000 seconds, for the mixtures tested), although at the cost of incurring efficiency penalties due to the velocity dispersion. It must be noted that this mixed regime had already been used in the earlier generation of Colloid Thrusters [4,5,6], but only under very high voltage conditions where multiple Taylor cones are forced to coexist on one site; this so-called "Highly Stressed Regime" yields relatively high thrust per site, but with lower efficiency and wide spray angles. By contrast, we have tried to stay within the single-cone regime, that yields better collimated and more readily controlled sprays. The lower thrust per emitter can in principle be made up by increasing the degree of multiplexing.

Several of the techniques developed in the initial phase were later found useful by other researchers in this field, in particular our use of ion optics to focus the sprays and allow longer path lengths for Time-of-Flight (TOF) mass spectroscopy. We also helped the AFRL Hanscom Laboratory in the initial setting up of the flow control and related

details for their Laboratory. In return, the AFRL expertise in ion detection and mass spectrometry has been invaluable to our own efforts.

Paper AIAA 2002-3814, by P. Lozano and M. Martinez-Sanchez [7] illustrates the following phase, where we began to generate new experimental data related to the mixed ion/droplet regime. Concentrated Formamide solutions were used, and the methods involved combined TOF (for charge/mass determination) and Stopping Potential energy analysis. The paper pointed out one source of apparent specific charge spread related to the use of planar, as opposed to spherical collecting surfaces. This effect was later put to use by Fernandez de la Mora for helping establish the angular distribution of a spray [8].

This line of research culminated in the Doctoral Thesis by Paulo C. Lozano [9], where the techniques are thoroughly explained and multiple results are presented and discussed. Formamide solutions with over 90% ion current fraction were obtained, but, as noted, no stable case was obtained with pure ions using this fluid. Results are also shown with the ionic liquid EMI-BF4, for which only pure-ion sprays were emitted at the lowest flow rates. The basic reason why ionic liquids emit sufficient ions to quench the jet formation process, while solutions of equal or higher conductivity do not, remains unexplained to this day, although it may relate to basic differences in the ion extraction free energy. The Thesis documents the nature of the emitted ions, that were found to be a mixture of "monomer" and "solvated" ions, with molecular masses of 111 and 309 amu (positive polarity).

Our work was put into context by the parallel and cooperative research at the AFRL in which we reported 89 and 298 amu (negative polarity) emission. The joint results are presented and discussed in paper AIAA-2003-4848 [10]. The fact that both polarities can be emitted with very similar extraction voltages and result in ions of similar masses is quite significant with a view to the design of Bipolar Thrusters, which can avoid the use of electron emitters as neutralizers.

Continuation of this basic work produced additional important results about the ionic regime: (a) Proof that External Wetting can be used in vacuum with Ionic Liquids to produce a much simplified feeding mechanism, akin to that used in Liquid Metal Ion Sources, and yielding streams of pure ions. (b) Proof that if the polarity of the spray is reversed at moderately low frequencies (of the order of one Hz), no electrochemical processes occur on the immersed counter-electrode. This latter result is essential to the use of these ion sources for propulsion, because if electrochemistry did occur (as it does under normal DC operation), the ionic species not extracted in the spray would be wasted in the propulsive sense, and might also foul or corrode the neutralizing surfaces. It is worth pointing out that this same effect is present in the traditional droplet regime or in the mixed ion/droplet regime, but as long as a solvent is present and droplets are emitted, it appears that the products of neutralization can be carried by the liquid away form the neutralizing surface, and eventually into the emitted droplets. These important results were later fully documented in several Conference and Journal papers (see Section 4 of this Report).

It became clear at that time that the new generation of thrusters made possible by these advances into the pure ion regime cannot be called "Colloid Thrusters", since no colloid is really present anymore. No clear name had as yet emerged, but after a discussion with colleagues during the 2nd Colloid Thruster/Nano Electrojet Workshop (MIT, April 14-15, 2005, Ref. [11]) an agreement was reached to designate both types of engines (with either charged droplets or ions) as "Electrospray Thrusters".

2. Microfabrication of Colloid/Electrospray Arrays (6/1/01-11/30/03)

A preliminary exploration of the concept of producing arrays of large numbers of individual colloid emitters by using modifications of the electronic industry's microfabrication techniques had been previously carried out at MIT with the support of the C.S. Draper Laboratory. The results were documented in two M.S. Theses, by M. Paine [12] and L.F. Velasquez [13], respectivley. The concept proposed by Paine involved micro-capillary channels oriented perpendicular to the face of a silicon wafer. An analysis by Velasquez concluded that this configuration could not provide the level of hydraulic resistance needed to stabilize operation of the emitters. Velasquez then proposed a design where a multiplicity of micro-channels are to be carved in one plane, half on each of two wafers which are eventually joined together. The details of this geometry, and a first cut at a fabrication schedule, are contained in his Master's Thesis[12]. With the award of this AFOSR Grant, the research proceeded to the actual fabrication phase, using the facilities in the MIT Microsystems Technology Laboratory (MTL), and with the active participation of Prof. A. Akinwande who has extensive microfabrication experience. It is worth mentioning here that the microfabrication processes involved in the construction of this engine are by no means standard, so that the process had to be itself designed in parallel with the engine.

The design and initial fabrication steps of a Linear Colloid Array were described in our first Yearly Report[1]. That design was further developed and then implemented during the following two years[2]. Many innovations were required along the way, due to the specialized requirements of the device. Particularly challenging were the processes to form the sharp "spouts" terminating each of the multiple capillary ducts, and also the attachment "clips" needed to support the extractor and accelerator electrodes. These electrodes had been initially designed to be integral with the engine substrate, but considerations of high-voltage insulation forced a change to separate structures, to be attached to the substrate with minimal contact, yet with high positional accuracy.

The design considerations and initial fabrication steps were presented in Ref. [14], presented at the 6th International Symposium: Propulsion for Space Transportation of the XXIst Century, (Versailles, France, 2002). A high hydraulic impedance was selected in order to make the flow rate per channel independent of downstream perturbations. This dictated a layout where the individual square channels (240, 12 μm each) were carved on the surface of one Silicon wafer (375 μm thick), and a second wafer was then bonded to it as a sealant. The ducts end at the edge of the wafers, and a special process was devised to sharpen each ending in two perpendicular directions, in order to reduce liquid spilling and to intensify the electric field at the tips. High conductivity Formamide

solutions were selected as the nominal working fluid, and operation in the droplet/ion regime was contemplated. Formamide is moderately volatile, but the very small duct diameter should reduce vacuum evaporation losses to under 10% of flow rate. The total thrust expected from the device is on the order of 20 μN, at a specific impulse between 500 and 1000 sec.

Further progress along these lines is described in the paper AIAA-2002-3810 [15]. In particular, the mechanical design of the electrode attachment clips is detailed, and evidence is shown of satisfactory bonding of the two wafers comprising the hydraulic system. In the paper AIAA-2003-4850 (Ref [16]), the final fabrication steps for the hydraulic system are shown, including partial spout sharpening, and the first test results are documented, using a test rig built for the purpose. The extraction and acceleration were accomplished by a detached planar electrode, and post-test examination of this electrode showed the impacts of individual spray jets and confirmed the expected separation and jet spreading angle (under 20°). Problems were encountered with flow control and fluid spillage, and further testing was planned to refine the start-up procedures and to produce hydrophobic external surfaces. The final results of this work on a Linear Colloid Array appeared in additional publications that are reviewed and referenced in Section 5.

After our basic physics research had proven the feasibility of external wetting as a feed method for vacuum operation using Ionic Liquids, we undertook preliminary fabrication work on methods to produce Silicon arrays for this purpose. The initial efforts are also presented in Ref. [16]. A method was devised in which the site of each of the eventual emitting tips is masked using a small star-shaped silicon oxide mask; a sequence of anisotropic DRIE cuts and isotropic undercutting steps then removes the unprotected material around the tips, and sharpens the remaining tall posts to the desired degree. Two generic morphologies were obtained; flat-topped posts with sharp polygonal contours ("volcanoes"), and single-tipped sharp "pencils", with tip radius of curvature under 1 μm. The posts are typically 300 μm tall, with 40 μm diameter, and can be spaced arbitrarily. Many sample arrays were produced with spacing between 2 mm and 200 μm. As produced, the Silicon surface is smooth and partially hydrophobic, but a Chlorine-based surface treatment was devised that roughens the surface and produces "Silicon grass" or "Black Silicon", with controllable protuberances on the order of 1 μm size. This extends to the post surfaces as well, and in tests, a single drop of EMI-BF4 ionic liquid placed anywhere on a 1 cm^2 test coupon was seen to spread in a few seconds and uniformly cover the coupon, including the sharpened posts. Subsequent test showed that these wetted tips can be made to emit what appears to be a pure ion spray (pending at that time TOF confirmation, which has more recently been obtained in unpublished 2005 work by Tanya C. Garza, of our Laboratory), thus opening the way for very compact and robust planar arrays for propulsion. These preliminary spraying results were reported in Ref [16] as well.

3. Theoretical Work (6/1/01-11/30/03)

Initial efforts towards the development of a fluid model for describing the Taylor Cone-Jet regime of operation were reported in our first Yearly Report[1]. This work was continued and further developed in the next two years. Paper AIAA-2002-3812 (Ref [17]) explains the formulation and methods of solution. A number of results were presented in Ref [18]. It is shown in [18] that the calculated current for a number of fluids and operating conditions closely matches the known empirical values reported by Fernandez de la Mora [19]. The axial profiles of the various quantities of interest are computed and displayed, yielding a wealth of physical information on the jet formation processes and influences. It is seen, for example, that the surface charge density remains in all cases within about 20% of its equilibrium value, even in the region of transition from the cone (where the current is conductive) to the jet (where conduction ceases and surface charge is carried by convection). Numerical experience shows that no stable solutions can be obtained when the flow rate is less than a certain value, which turns out to be of the order found for the experimental minimum flow as well. A discussion is included in Ref. [18] to show that the liquid conductivity remains constant along the flow, and that depletion of the counter-ions is never approached. Additional results and further extension of the parametric domain of numerical applicability of the model were obtained in the final year of the Grant, and are discussed and referenced in our Section 6.

4. Basic Physics (12/1/03-12/31/04)

In the final year covered by this grant, a substantial amount of work has been done in terms of advancing our fundamental understanding of electrospray emissions in the regimes of interest for space propulsion. These advances were demonstrated in a number of experimental tests, which in turn have motivated further theoretical work, while at the same time influencing the design and fundamental working principles of the proposed microfabricated electrospray thrusters. These advances have been carefully documented in detail in a series of journal and conference publications, which are included in the present report. The following paragraphs contain a summary of last year's activities. The reader is encouraged to look at the cited references.

As discussed above, we had demonstrated in the early part of this project that interesting electrospraying regimes can be achieved using organic solvents, such as salt-doped formamide. In particular it was found that under the right conditions (low flow rate, high liquid conductivity) ion emission is possible using standard capillary-type emitters[20]. This introduces a way of obtaining higher specific impulses at the expense of lower propulsive efficiencies. Also, it was found back then that the efficiency problem can be reduced significantly using ionic liquids (or room temperature molten salts), since pure ion emission (and therefore high Isp) can be obtained, at least for the ionic liquid EMI-BF4. In addition to this, ionic liquids have negligible vapor pressure thus reducing concerns about evaporative losses in the vacuum of space, they are also stable in a wide range of temperatures, they have relatively low viscosities, high conductivities and moderate surface tensions.

The properties of these liquids allow them to be used in an external architecture similar to liquid metal ion sources (LMIS). Our group was the first to demonstrate that ion emission can be obtained from solid emitters externally wetted with an ionic liquid[21]. The emitters were made out of tungsten wire, electrochemically treated to increase wettability and etched to produce a fine tip (~10 μm radius of curvature) from which ions are electrostatically evaporated after applying a high voltage between the wire and an extraction electrode placed near the wire tip. The properties of these ions are remarkable in terms of their energetic spreading (only a few eV). Also, their kinetic energy after full acceleration corresponds nearly to the applied voltage (a few eV below the applied potential). These properties allow the beams to be electrostatically focused with very little losses. We also found that the angular spreading of the ion beams is well contained within a 20° half angle from the centerline. This is important, since angular spreading reduces the thrust efficiency[22] and also has a deep impact on the thruster design if ion interception by the extractor-accelerator electrodes is to be avoided.

On the other hand, a generic problem in working with electrosprays in the pure ionic regime was uncovered: as one of the polarities is being extracted from the source, the opposite charges will accumulate on the counter-electrode, which in the case of the tungsten wire emitters is the metal surface. This charge accumulation increases the voltage difference across the metal-liquid interface, eventually becoming large enough so that strong electric fields develop and electrons are transferred from the liquid to the metal, or vice-versa, i.e., electrochemical reactions. These reactions have some effect on the neutralized species or the emitter substrate. In the case of EMI-BF4, extraction of EMI positive ions leave the substrate with an excess of BF4 negative ions that when neutralized react to produce a dielectric layer which eventually destroys the electrical contact between the metal and the liquid, and therefore no more charge can be extracted from the source. It is interesting to note that this problem happens in capillaries as well when working in the pure ionic regime: in this case, the reaction products accumulate gradually over the capillary walls, and may eventually clog the small tubes. In contrast, the electrostatic extraction of BF4 negative ions produces an accumulation of positive EMI ions on the electrode, the reaction products of which are released in gas form, apparently without damaging the substrate or the liquid integrity. Regardless of the outcome, however, the appearance of electrochemical reactions represents a serious problem in this kind of thrusters. A way to solve it was proposed by our team[23]. The technique takes advantage of the high electrochemical window (or maximum double layer voltage before electrochemistry occurs) of ionic liquids (~4-6 V) and the relatively large interfacial surface area. The double layer behaves like a capacitor with a time constant proportional to these two quantities (and inversely proportional to the extracted current and layer thickness). If the voltage polarity is reversed before the capacitor charges to the limit, then no reactions occur. This forces the ion source to operate in an alternating mode at a frequency dictated by certain dynamic limitations of the system. For our case, the frequencies are of the order of 1 Hz, which is technologically advantageous since the combination of high voltages and high frequencies is undesirable.

The emission of positive and negative charges can be used to produce charge neutralization without the use of an external cathode, which typically limits the life of

many electric propulsion devices[24]. More work needs to be done in this respect, but preliminary tests show that this technique can achieve neutralization, for example, with a group of emitters working with a given polarity while a different group fires simultaneously opposite charges, alternating their charge roles from time to time to avoid electrochemical reactions, as discussed above.

The introduction of the alternation technique solves the electrochemistry problem, but introduces a dynamic response every time the emitters are re-started. The main dynamic characteristic in the response comes in the form of temporal delays between the voltage onset and the appearance of ionic current[25]. These delays are therefore the limiting factor in the alternation frequency, since no current could be emitted if the delays were to become of the same duration as the alternation half period. The delays, however, are not fixed but depend on operational and design characteristics. Our results indicate that viscous flow ultimately determines the response time (Taylor cone formation) of the liquid. This is consistent with other dependencies, such as the linear increase of emitted current with temperature (viscosity in an ionic liquid decreases almost linearly with temperature). In any case, for the tungsten emitters, typical delay values range between 5-10 ms. This allows practical alternation frequencies up to 100 Hz, which is much higher than required for our emitters. Still to be resolved are the frequency requirements and a detailed characterization of the dynamic response for microfabricated emitters.

Additional work was also done to test the emission of cone-jets from flat dielectric surfaces[26]. This configuration is very simple to manufacture and consists of a dielectric non-wetting material in which at least one small ID hole is drilled. Liquid is forced from a reservoir on one side of the hole while an extractor plate is located downstream from the other side. A high voltage is applied between the liquid reservoir and the extractor and a cone-jet forms at the base of the hole. The non-wetting property of the material effectively anchors the cone to the hole rim, while the low dielectric constant value (for Teflon, $\varepsilon \sim 2$) simulates a vacuum environment surrounding a fine liquid column, which therefore acts as a conductive needle and provides its own electric field enhancement. Thus, the voltages required to produce emission are comparable to those of metallic needles. Massive arrays of holes can be microfabricated quite easily, hence the attractiveness of this option.

5. Micro-fabrication (12/1/03-12/31/04)

Our previous microfabrication efforts were centered on producing an internally fed engine with a series of high aspect ratio capillary tubes laid out side-to-side over a Silicon wafer. A series of fabricating techniques were created and improved for this design. The bulk of this work is described in L.F. Velasquez-Garcia's doctoral thesis[27] , conference papers[28], and a journal paper under review[29]. The main idea behind the linear design is to exploit the high aspect ratio of the individual emitters to create enough viscous resistance to liquid flow such that there is no interference (pressure gradients) between the different emission sites. The engine was designed to operate in the cone-jet regime sand was tested with doped formamide. Preliminary results indicated uniform droplet emission with the total current following the square-root law dependence on flow rate as

established in the electrospray literature. The engine incorporated a novel design for the extractor using clip-like structures, which could be manually assembled. These techniques may prove useful in other microfabrication applications.
Given the new developments with the tungsten emitters we decided to shift the focus of our microfabrication effort towards exploiting the properties of ionic liquids and the advantages of the externally wetted configuration. A critical problem in our research was finding a way to make the ionic liquid wet the Silicon substrates since initial studies demonstrated very poor wetting. A first (and ultimately very good) solution to the problem was found by introducing some artificial roughness to the Silicon surface. These treatments involve reactive plasma bombardment at some particular conditions[30], creating the so-called *black Silicon,* in effect producing some degree of surface roughness, which can transport and distribute liquid quite efficiently.
In addition, the hydraulic resistance to flow is now provided by the substrate itself, so there is no need for large aspect ratio tubes. The main issue was to produce wettable, point-like structures protruding from the Si substrate from which ion emission can occur. Variations of relatively simple microfabrication techniques were used to produce many types of emitters, from the point-like *pencils* to the wider *volcanoes*. Initial testing of these devices indicates that ion emission is possible and that the beam characteristics are in general similar to those produced by the tungsten emitters[31] . Still to be resolved is the coupling of a microfabricated extractor to the emitter substrate. This task becomes complex since the clearances are very small and there is a need to isolate electrically and hydraulically both structures.

6. Theory (12/1/03-12/31/04)

Our numerical modeling tools were improved in the final year of the contract. The main product is the software described in Carretero's PhD thesis [32] and in several conference publications [33,34] . The numerical model represents a first attempt to obtain a complete description of the cone-jet formation mechanism using a quasi-1-D time marching algorithm, solving the continuity and momentum equations with viscous and electric terms. The model results were able to reproduce experimental measurements in terms of the current emitted as a function of flow rate for a variety of liquids. In particular, the dependence of current on dielectric constant is reproduced reasonably well. The model also allowed to verify whether some scaling laws using in the theory are more appropriate than others and gave estimates of the energy losses by different mechanisms, like ohmic dissipation, surface energy and viscous losses. The main difficulty with this model was the computational stiffness when using liquid solutions with high conductivities since the time step scales as the electric relaxation time, which can be very small at $K \sim 1$ Si/m. This, in turn, made it difficult to estimate the emissions of ions from the liquid surface in order to simulate the observations of highly conductive solutions at low flow rates, that show copious ion emission in experiments. A preliminary fix to the problem involved reducing artificially the energy barrier for ion emission, but this produced counterintuitive results. A modified version will be required to cope with these issues in the future.

In addition to the electrohydrodynamic model, a particle-in-cell code was developed to track the trajectories of ions and droplets [34] . The code assumed different kinds of initial and boundary conditions to study the effect of space charge on beam spreading. This kind of modeling needs to be extended to incorporate the jet break-up region (to have more accurate initial conditions) and to include particle-particle interactions such as droplet collisions, coulombic explosions and ion capture.

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